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(54) GELLING OR SOLIDIFYING AGENT FOR ORGANIC LIQUID

(57)Abstract:
PURPOSE: To obtain a gelling or solidifying agent for organic liquids useful as lubricants, releasants or adhesives, and capable of producing uniform, smooth, viscous and stable gelled or solidified products, containing, as an active ingredient, a specific transesterification product.
CONSTITUTION: This gelling or solidifying agent contains, as an active ingredient, an transesterification product obtained by transesterification between a triglyceride with a 2–28C straight chain saturated fatty acid as the constituent fatty acid and a diester of a 20–28C aliphatic saturated dibasic acid and a lower alcohol such as a 1–4C straight chain or branched alcohol. This transesterification product at least contains such a partial transesterification product that one of the ester linkages between said aliphatic saturated dibasic acid and the lower alcohol is left intact.

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CLAIMS

[Claim(s)]
[Claim 1] Gelation or the solidification agent of the organic liquefied object which becomes considering said ester exchange reaction product which contains at least the partial ester exchange reaction object which made one side remain among the ester bonds of said aliphatic series saturation dibasic acid and lower alcohol which carry out the ester exchange reaction of the triglyceride which uses the straight chain-like saturated fatty acid of carbon numbers 2-28 as a configuration fatty acid, and the lower alcohol diester of the aliphatic series saturation dibasic acid of carbon numbers 20-28, and are obtained as an active principle.
[Claim 2] The gelation according to claim 1 or the solidification agent whose lower alcohol is the shape of a straight chain and side-chain-like alcohol of carbon numbers 1-4.
[Claim 3] The gelation according to claim 1 or the solidification agent whose carbon numbers of straight chain-like saturated fatty acid are 18-28 when the carbon number of an aliphatic series saturation dibasic acid is 20.
[Claim 4] The gelation according to claim 1 or the solidification agent whose carbon numbers of straight chain-like saturated fatty acid are 2-28 when the carbon number of an aliphatic series saturation dibasic acid is 28.
[Claim 5] The gelation according to claim 1, 2, 3, or 4 or the solidification agent which is the alcohol with which an organic liquefied object presents the shape of liquid in ordinary temperature, a fatty acid, ester, the ether, or a hydrocarbon.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Industrial Application] This invention relates to the ester exchange reaction product which gels or solidifies the liquefied organic substance in ordinary temperature. Gelation or the solidification agent of this invention can be used in the processing fields, such as the electrical and electric equipment, an electron, the device for MAG, a machine, an automobile, the general cargo for days, a color, ink, a coating, cosmetics, toiletries, drugs, agriculture, fishery, feed, the food field and paper, fiber, leather, resin, a macromolecule, rubber, and a metal, etc.
[0002]
[Description of the Prior Art] Conventionally, the metal soap of (1) higher fatty acid, 12-hydroxy stearin acid, a JIBEN zylidene sorbitol, JIBEN zylidene xylitol, a N-acylamino acid derivative, (2) dextrin fatty acid ester, an acrylic-acid system polymer, etc. are known as what has the function which solidifies fats and oils, a hydrocarbon, or a solvent to gel.
[0003] Among these, the type of (1) solidifies the whole to gel by making it dissolve or distribute in liquefied ester and fats and oils, and mainly cooling to homogeneity, at them. this gelling agent -- melting point [of 12-hydroxy stearin acid]: -- 80 degrees C and melting point:160 degree C of a JIBEN zylidene sorbitol -- as -- generally, the melting point was high, and in solidifying the so-called organic liquefied objects, such as fats and oils, for this reason, it needed to perform heating actuation beforehand, and to dissolve the gelling agent itself, or to carry out melting of for example, a gelling agent and the fats and oils. Moreover, to organic solvents, such as general-purpose isoparaffin, a hexane, and ethanol, it dissolved industrially as a solvent of a low-boiling point, and it begins by adding very a lot of gelling agents, and can solidify, and this type of gelling agent was unsuitable to solidification of this organic solvent.
[0004] On the other hand, although there is "an AKUA rucksack CA" by Nippon Shokubai Kagaku Kogyo Co., Ltd., for example as an acrylic-acid system polymer as a gelling agent of the type of (2) and this solidifies almost all the hydrocarbons system compound, to the fats and oils which have an ester system compound and alkyl group chain length's large higher-fatty-acid residue, there is especially no **** about the effectiveness of gel solidification. And this type of thing was difficult for obtaining a uniform gel object, in order to make the so-called gelled object absorb in a gelling agent and to solidify to the things of the aforementioned (1) type being melting and a thing to solidify about a gelling agent and a gelled object.
[0005]
[Problem(s) to be Solved by the Invention] Therefore, the object of this invention is little addition about organic liquefied objects, such as alcohols which present the shape of liquid in ordinary temperature, ester, and hydrocarbons, and is to offer homogeneity and a smooth solid-state-like object, a gel compression object and the gelation to make, or a solidification agent preferably.
[0006]
[Means for Solving the Problem] In order to attain said object, this invention persons came to complete a header and this invention for a gel object or a compression object being obtained by using a specific ester exchange reaction product, as a result of repeating examination wholeheartedly. Namely, the triglyceride with which this invention uses the straight chain-like saturated fatty acid (only henceforth a fatty acid) of carbon numbers 2-28 as a configuration fatty acid, Carry out the ester exchange reaction of the lower alcohol diester of the aliphatic series saturation dibasic acid (only henceforth a dibasic acid) of carbon numbers 20-28, and are obtained. It is gelation or the solidification agent of the organic liquefied object which becomes considering said ester exchange reaction product which contains at least the partial ester exchange reaction object which made one side remain among the ester bonds of said dibasic acid and lower alcohol as an active principle.
[0007] One of the indispensable raw material components for manufacturing the ester exchange reaction product of this invention is a triglyceride with which it consists of straight chain-like saturated fatty acid of 2-28, the triglyceride, i.e., the carbon number, of a specific fatty acid. An acetic acid, a propionic acid, a caproic acid, a caprylic acid, a capric acid, a lauric acid, a myristic acid, a palmitic acid, stearin acid, 10-hydroxy stearin acid, 10-keto stearin acid, 12-hydroxy stearin acid, arachin acid, behenic acid, a montanoic acid, etc. can be raised as an example as concrete straight chain-like saturated fatty acid, and in this invention, even if it uses it as a triglyceride of independent [these] or mixture, it does not interfere.
[0008] this triglyceride -- a conventional method -- an esterification reaction with a glycerol -- it can obtain -- moreover, heavens -- a product -- hydrogenation, judgment, etc. are processed with the thing of the origin, for example, soybean oil, oleum rapae, olive oil, cotton seed oil, the linseed oil, castor oil, palm oil, palm oil, fish oil, beef tallow, lard, etc., and it may be obtained. An inside chain saturated fatty acid triglyceride and the so-called MCT are suitable for this invention. In addition, as long as the unsaturated fatty acid as a glyceride component, a side-chain-like fatty acid, diglyceride, a monoglyceride, etc. are amounts a little, they may be contained.
[0009] moreover -- as the lower alcohol diester of the dibasic acid which is another raw material component -- a carbon number -- 20-28 -- it needs that it is JIESUERU with the shape of a straight chain of 1-3, and side-chain-like alcohol more preferably [it is desirable, and the aliphatic series saturation dibasic acid of 22-28 and a carbon number are desirable, and] than 1-4. The thing or carbon number of partial saturation cannot obtain [a dibasic acid] easily the dibasic acid to which, as for the ester exchange reaction product of less than 20 thing, a carbon number exceeds 28 by gelation ability falling as a industrial raw material. therefore, independent [in dibasic acids such as eicosa dicarboxylic acid, a docosa KOSAJI carboxylic acid, tetracosa dicarboxylic acid, hexacosa dicarboxylic acid, and OKUTAKOSA dicarboxylic acid] in this invention -- or OKUTAKOSA dicarboxylic acid can be easily isolated from oil seeds including the Goma seed, and is [that what is necessary is just to use it, mixing] suitable. Said dibasic acid diester-izes this with lower alcohol, such as a methanol, ethanol, propanol, isopropanol, and a butanol, with a conventional method, and it is used for it.
[0010] Although the above mentioned configuration fatty acid and above mentioned dibasic acid of a triglyceride can be used in this invention, combining them suitably, when the carbon number of a dibasic acid of especially desirable combination is 20, the carbon numbers of the configuration fatty acid of a triglyceride are 18-28, and when the carbon number of a dibasic acid is 28, the carbon numbers of the configuration fatty acid of a triglyceride are 2-28.
[0011] What is necessary is just to adopt either of the approaches described below, in order to obtain the ester exchange reaction product of this invention using said raw material. That is, it mixes at the rate of a compounding ratio of less than two mols preferably less than ten mols of triglycerides to one mol of dibasic-acid diester so that it may mention later, and the chemical ester exchange reaction of a triglyceride and the dibasic-acid diester is carried out, using metal alcoholates, such as a sodium METOKI side and lithium ethoxide, as a catalyst, or an enzyme-ester exchange reaction is carried out, using lipase as a catalyst.

[0012] Among these, the ester interchange method by lipase has 1 and a desirable ester interchange method using the lipase which has selectivity in the 1 or 3rd place in this invention although the 2 or 3rd place is divided into a selection mold the 1 or 3rd place with a random mold from the difference of the singularity over the location of a glyceride. As this type of selectivity lipase, the thing of the microorganism origin is highly convenient, and they are Rhizopus delemar (Rhizopus delemar) and Mucor. An I high (Mucor miehei), Aspergillus The thing from nigre (Aspergillus nigar) etc. can be used and it is especially Alcaligenes. The lipase of the ESUPI (Alcaligenes sp.) origin, for example, the Meito Sangyo [Co., Ltd.] make, a trade name: Lipase QL is suitable.
[0013] What is necessary is for an ester exchange reaction to be in anhydrous thru/or the coexistence condition of the moisture of ultralow volume substantially, when using lipase, and to be a non-solvent preferably and just to stir it at 20-130 degrees C under existence of a solvent inactive for a reaction, or nonexistence, for about 10 minutes to 100 hours that what is necessary is just to stir a raw material at 80-140 degrees C as an anhydrous condition substantially for about 0.5 to 5 hours when using for example, a metal alcoholate. In addition, since said lipase QL discovers ester interchange activity at the elevated temperature of about 80-130 degrees C, it can be used very conveniently at the reaction using a raw material high-melting [like this invention].
[0014] Progress of said reaction can be evaluated by measuring the content in the system of reaction of the lower alcohol ester of the fatty acid which carries out a byproduction with a gas chromatography, and, thereby, should just determine the termination event of a reaction. Since the lower alcohol ester of the fatty acid which an unreacted raw material component may remain in an ester exchange reaction object, in addition carries out a byproduction is intermingled, if separation clearance is carried out and well-known approaches, such as rinsing, alkali deoxidation, solvent separation, vacuum distillation, an adsorbent, and molecular sieving, take these further, decolorization and deordorization processing will be performed and refined.
[0015] The ester exchange reaction product of this invention obtained in this way The component in which the triglyceride and dibasic-acid diester of a raw material carried out the ester exchange reaction at a rate of 1:1, 1:2, and 2:1 by the mole fraction is used as a major component. In addition, it is the mixture with a melting point of about 40-90 degrees C with which said raw material contains monochrome thru/or the component by which oligo esterification was carried out the shape of a straight chain, and in the shape of a mesh. And the ester interchange only of one ester bond is carried out among dibasic-acid diester, and it is characterized by containing at least the component which remains while another side has been lower alcohol ester and by which the partial ester exchange reaction was carried out so to speak. In order to make with such a presentation, less than ten mols of triglycerides are preferably set as less than two mols for the rate of a raw material compounding ratio of an ester exchange reaction to one mol of dibasic-acid diester. About an organic liquefied object, it gels, or is hard to solidify and the ester exchange reaction product which does not contain said partial ester exchange reaction component becomes.
[0016] In addition, the check of the ester bond of a dibasic acid and lower alcohol remaining in the ester exchange reaction product of this invention can carry out hydrochloric-acid hydrolysis of what performed said purification processing and removed the unreacted object and the by-product, for example, and can be performed by analyzing the lower alcohol generated in a decomposition product with a gas chromatography.
[0017] the ester exchange reaction product of this invention is independent in this — or it can mix and can make with gelation or the solidification agent of an organic liquefied object. Although an organic liquefied object says the organic compound which presents a liquid condition in ordinary temperature here and an example is shown below, this invention is not limited to these. First A methanol, ethanol, isopropanol, a butanol, ethylene glycol monoethyl ether, Ethylene glycol monobutyl ether, the butylene-glycol monoethyl ether, 2-ethylhexanol, nonanol, 2-heptyl undeca Norian, 2-octyl dodecanol, oleyl alcohol, ethylene glycol, It can be aimed at fatty acids, such as the alcohols of the shape of the shape of a straight chain, such as dipropylene glycol and a glycerol, and a side chain, saturation, or partial saturation, an acetic acid, a propionic acid, a caproic acid, a caprylic acid, oleic acid, and various isostearic acid.
[0018] Moreover, hydrocarbons, such as ether, such as fats and oils, such as ester, such as myristic-acid isopropyl, myristic-acid octyldodecyl, a Tori 2-ethyl hexanoic-acid glyceride, a mixed medium-chain-fatty-acid glyceride, and ethyl acetate, soybean oil, oleum rapae, sunflower oil, safflower oil, cotton seed oil, olive oil, sesame oil, linseed oil, and fish oil, and these partial decomposition products, wood ether, diethylether, and the petroleum ether, a pentane, a hexane, a heptane, an isooctane, paraffin, and isoparaffin, are suitable. Liquefied silicon oil, kerosene, etc. can be made into an object in the ordinary temperature other than these.
[0019] if it adds 0.5 to 3% of the weight and the ester exchange reaction product of this invention is preferably required 0.1 to 10% of the weight to said organic liquefied object — about 80 degrees C — warming — after melting — light — stirring — ordinary temperature — **** — it is — if it cools and puts on about 5 degrees C gently, it will be uniform and a smooth gelation object or a smooth compression object with viscosity nature thru/or a gel compression object will be obtained. The whole system holds a homogeneity condition, without this thing generating a liquid part at the temperature of about 40 degrees C or less.
[0020] In addition, gelation or the solidification agent of this invention may blend independent or the solid-state fat which becomes this from glycerides, such as other palmitic acids which are a candelilla wax, waxes, for example, carnauba wax, with conventionally still better known optimum dose, a montan wax, a micro crystallin wax, paraffin wax, etc., stearin acid, and behenic acid, unless it deviates from the object of this invention, although only mixture does not interfere of said ester exchange reaction product. Moreover, you may use together with said well-known gelling agent.
[0021]
[Example] In the following synthetic examples and examples, % is weight criteria.
It is inside chain saturated fatty acid triglyceride (Nisshin Oil Mills, Ltd. make, trade name:ODO, and configuration fatty acid are 75% [of caprylic acids], and 25% of capric acids) 49.4g (0.1 mols) to the flask furnished with synthetic example 1 agitator and a thermometer, The approach of a Japanese Patent Application No. [for which these people applied previously / No. 230734 / five to] publication, i.e., the settlings of the Goma crude oil, (cage) is distributed to ethanol. Make it dissolve, and cool and dimethyl ester 49.6g (0.1 mols) of the OKUTAKOSA dicarboxylic acid obtained by separating the insoluble matter which deposits is used as a raw material. Alcaligenes The lipase (the Meito Sangyo Co., Ltd. make, lipase QL) of the ESUPI (Alcaligenes sp.) origin is added 1% of pair raw materials. Stirring at 90 degrees C, the gas chromatography analyzed the content of the fatty-acid methyl ester which carries out a byproduction, and the ester exchange reaction was performed for 72 hours until the increment was no longer accepted. after reaction termination and a silica gel column chromatography — and vacuum distillation processing was carried out, the ester exchange reaction object was refined, and 82.5g (sample notation: referred to as A) of ester exchange reaction products of this invention was obtained. this thing — acid-number: — they were 4.7, hydroxyl value:3.4, and melting point:57-64 degree C.
[0022] Dissolve the above-mentioned ester exchange reaction product in a lot of tetrahydrofurans, and present the high performance chromatography using the column filled up with polystyrene divinylbenzene system polymer gel (polymer laboratory company make, trade name-L-gel). When molecular-sieving processing was performed and the quantum of the presentation was carried out, the component the inside chain saturated fatty acid triglyceride of a raw material and OKUTAKOSA dicarboxylic acid dimethyl ester carried out [the component] the ester exchange reaction at a rate of 1:1, 1:2, and 2:1 by the mole fraction is about 2:3:2, and these three components formed 83% of the whole. The remaining components were what is considered that both raw materials repeated the ester exchange reaction. Moreover, the methanol was detected, when the above-mentioned ester exchange reaction product was hydrolyzed with the conventional method using 1N-hydrochloric acid and the gas chromatography was presented with the decomposition product. It checked that methyl ester remained in the above-mentioned ester exchange reaction product by this.
[0023] The ester exchange reaction of behenic acid triglyceride 114g (0.1 mols) and the dimethyl ester 37g (0.1 mols) of eicosa dicarboxylic acid which carried out ester composition with synthetic example 2 conventional method was carried out like the synthetic example 1, the silica

gel column chromatography was presented with the reactant, separation clearance of the impurity (an unreacted raw material and by-product) was carried out, and 92.4g (sample notation: referred to as B) of ester exchange reaction products of this invention was obtained. this thing -- acid-number: -- they were 3.2, hydroxyl value:2.9, and melting point:57-61 degree C.

[0024] In the example 1 of synthetic example 3 composition, lipase 1% was replaced to sodium METOKI side 0.5%, the chemical random mold ester exchange reaction was performed for 30 minutes at 80 degrees C under reduced pressure, purification processing of the reactant was carried out similarly, and 79.8g (sample notation: referred to as C) of ester exchange reaction products of this invention was obtained. this thing -- acid-number: -- they were 0.7, hydroxyl value:1.8, and melting point:58-64 degree C.

[0025] The synthetic example 4 (example 1 of comparison composition)

in the synthetic example 1, using triglyceride (Wako Pure Chem make trade name: triacetin) 109g (0.5 mols) and methyl ester 142g (0.5 mols) of tetra-deca dicarboxylic acid (NIKKO KYODO Make and trade name:tetradecane JIOIKKU -- acid) which uses an acetic acid as a configuration fatty acid as a raw material, the ester exchange reaction was carried out similarly, purification processing was performed, and 182g (sample notation: referred to as D) of ester exchange reaction products was obtained. this thing -- acid-number: -- it was 4.9 and hydroxyl value:5.0 and was liquefied in ordinary temperature.

[0026] The gelation ability to the ester exchange reaction product (sample notation: A-D) obtained in the examples 1-4 of example 1 composition, 12-hydroxy stearin acid, and the soybean oil of carnauba wax was examined. The result is shown in a table 1. In addition, after heating and fusing the examining method at 80 degrees C, taking and stirring soybean oil and 3% of each of its sample to a beaker and cooling radiationally in ordinary temperature as it is for 1 hour, the condition of the obtained gel object was observed. assessment -- O: -- a hard, uniform, and smooth gel object and O: -- a uniform and smooth gel object and **: -- the part considered as what carries out solid liquid separation, and the thing of which x:compression is not done.

[0027]

[A table 1]

表1　大豆油のゲル化又は固化物

	本発明例			比較例		
添加物	試料A	試料B	試料C	試料D	12-ヒドロキシステアリン酸	カルナウバワックス
固形物の状態	◎	◎	◎	×	◎	×

[0028] It became clear that it forms a uniform gel compression object to soybean oil although the ester exchange reaction product (sample notation: A-C) of a table 1 to this invention is little addition compared with a conventional gelling agent and a conventional wax. Moreover, when these were saved for one month in ordinary temperature, what added the esterification product of this invention was maintaining the homogeneity condition, and the stable thing was accepted. In addition, although 12-hydroxy stearin acid made soybean oil solidify, the solid was hard and it was very a BOSO ****** feel, and the original solid was not reproduced when this was ******(ed) one time. On the other hand, the ester exchange reaction product of this invention is very smooth, has viscosity nature, and even if it crushed this, it returned to the original gel state easily. Sample D was inferior in gelation ability.

[0029] The gelation or solidification ability to the various organic liquefied objects of the ester exchange reaction product (sample notation: A) obtained in the example 1 of example 2 composition was investigated by the same approach as an example 1. The result is shown in a table 2. In addition, the sign of assessment is the same criteria as an example 1. From a table 2, to various liquefied objects, the ester exchange reaction product of this invention was little, had compression ability, moreover presented description with viscosity nature uniform [a solid] and smooth, and found that this property was effective to the broad polar substance.

[0030]

[A table 2]

表2　各種有機液状物のゲル化又は固化物

有機液状物	試料Aの添加率と固形物の状態	
	1 %	3 %
イソパラフィン	○	○
流動パラフィン	◎	◎
イソノナン酸イソノニル	○	○
トリイソオクタン酸グリセリル	◎	◎
リンゴ酸ジイソステアリル	○	◎
トリイソステアリン酸ジグセリル	○	◎
ジイソステアリン酸ジグセリル	○	◎
モノイソステアリン酸ジグセリル	○	◎
ヒマシ油	○	◎
大豆油	◎	◎
オレイン酸	○	○
灯油	×	○
エタノール	△	○
n－ブタノール	△	○
1，3－ブタンジオール	△	○

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[0031] According to the approach of the example 1 of example 3 composition, various ester exchange reaction products (sample notation: 1-11) were compounded, it added 3% to oleum rapae, and gelation or solidification ability was investigated. The result is shown in a table 3. In addition, the sign of assessment is the same criteria as an example 1. It became clear that desirable gelation or a solidification agent can be prepared from a table 3 by combining suitably the class of the configuration fatty acid of a triglyceride and dibasic acid.

[0032]

[A table 3]

表3　各種エステル交換反応生成物のゲル化又は固化能

	試料番号	エステル交換反応生成物の原料				固形化物の状態
		トリグリセリド		二塩基酸ジエステル		
		構成脂肪酸の種類[1)]	モル比	二塩基酸ジエステルの種類[2)]	モル比	
本発明例	1[2)]	C8/C10=75/25	1.0	C28ジメチル	1.0	◎
	2	C8/C10=75/25	1.0	C28ジプロピル	1.0	◎
	3[3)]	C22	1.0	C20ジエチル	1.0	◎
	4	C22	1.0	C28ジメチル	1.0	◎
	5	C 2	1.0	C28ジメチル	1.0	○
	6	C18	1.0	C20ジメチル	1.0	○
	7	MR－60[5)]	1.0	C20ジメチル	1.0	○
	8	C8/C10=75/25	0.3	C28ジメチル	1.0	◎
	9	C8/C10=75/25	1.0	C28ジメチル	0.3	○
比較例	10[4)]	C 2	1.0	C14ジメチル	1.0	×
	11	C22	1.0	C14ジメチル	1.0	×

Note One carbon number was expressed as C numeric value. For example, as for C22 of a fatty acid, C28 of a HEBEN acid and a dibasic acid is OKUTAKOSA dicarboxylic acid.

2) It is the same as the sample notation A.

3) It is the same as the sample notation B.
4) It is the same as the sample notation D.
5) high — erucic acid seed pole hard oil (Miyoshi Oil & Fat Co., Ltd. make): — fatty acid composition — a palmitic acid (4.7%), stearin acid (74.6%), arachin acid (5.0%), and behenic acid (15.7%)

[0033]

[Effect of the Invention] According to this invention, let said ester exchange reaction product which contains at least the partial ester exchange reaction object which it is [object] the ester exchange reaction product of the triglyceride which uses the straight chain-like saturated fatty acid of carbon numbers 2-28 as a configuration fatty acid, and the lower alcohol diester of the aliphatic series saturation dibasic acid of carbon numbers 20-28, and made one side remain among the ester bonds of said aliphatic series saturation dibasic acid and lower alcohol be an active principle. The becoming gelation or the solidification agent is obtained and homogeneity and the stable gelation which is smooth and has viscosity nature, or a compression object can be formed only by carrying out little addition of this at the ester which presents the shape of liquid in ordinary temperature, fats and oils, hydrocarbons, a polarity, a nonpolar organic solvent, etc. Therefore, the ester exchange reaction product of this invention can be made with the gelation or the solidification agent of an organic liquefied object which was excellent in the gelation ability of the low melting point compared with the conventional thing, and this gelation or a solidification agent can be effectively utilized as a solvent, the processing agent of oils, lubricant, a release agent, adhesives, a binder, a sealing agent, lubricant, the coating, a paint film agent, a volatile constituent modifier, etc.

[Translation done.]